EXTRACTION OF NIGELLA SATIVA USING MODERN HYDRO DISTILLATION TECHNIQUE

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A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering

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NOVEMBER 2006

DECLARATION

"I declare that this thesis is the result of my own research except as cited references.
The thesis has not been accepted for any degree and is concurrently submitted in candidature of any degree."

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In the memory of my dear mother Siti Rahani Bt. Amat

ACKNOWLEDGEMENT

In preparing this thesis, I was in contact with many people, researchers, academicians and practitioners. They have contributed towards my understanding and thoughts. In particular, I wish to express my sincere appreciation to my supervisor, Mr. Ahmad Ziad Bin Sulaiman for encouragement, guidance, critics and friendship. I am also very thankful to all the Teaching Engineers for their guidance, advice and also motivation. I am also indebted to FKKSA lectures for their guidance to complete this thesis. Without their continued support and interest, this thesis would not have been the same as presented here.

My fellow postgraduate students should also be recognized for their support. My sincere appreciation also extends to all my colleagues especially Mr. Nazeri B. Nasaruddin and other who have provided assistance at various occasions. Their views and tips are useful indeed. Unfortunately, it is not possible to list all of them in this limited space. I am grateful to all my members in KUKTEM.

ABSTRACT

Nigella Sativa (Jintan Hitam) can produce essential oil that is important in medicine and also important in developing our agriculture sector nowadays. To obtain *Nigella Sativa* essential oil, entrepreneur and researcher nowadays use hydro distillation method. So the objective of this study is to obtain essential oil from *Nigella Sativa* plant source using modern hydro distillation technique because of it market values, by investigating and understanding hydro distillation extraction process. For this research, the rotary evaporator will be use and the parameters which are expected to dominate in producing high yield of *Nigella Sativa* oil was the effect of time and the surface area of the Nigella Sativa seeds expose to the extraction process. The temperature for the extraction process is maintained at 100 ^{o}C and 1 bar for the pressure. It is expected that the optimum operating time will be establish from the experimental result. The main compound of the essential oil is Thymoquinone, which are around 50% from overall compounds.Therefore, the presence this compound should take as a characteristic for the essential oil and the essential oil will analyze using High Performance Liquid Chromatography (HPLC).

ABSTRAK

Jintan Hitam (*Nigella Sativa*) dapat menghasilkan pati minyak yang amat penting dalam bidang perubatan dan dalam bidang agrikultur pada hari ini. Untuk mendapatkan pati minyak tersebut, para usahawan dan penyelidik pada hari ini telah menggunakan kaedah penyulingan berair. Objektif kajian ini ialah untuk mendapatkan pati minyak disebabkan oleh harga pasaran yang tinggi. Ini dapat dilakukan dengan mengenalpasti dan memahami kaedah penyulingan berair. Untuk menjalankan eksperimen pada kali ini, alat yang akan digunakan ialah Peruap Berputar (rotary evaporator). Parameter yang dijangka akan mendominasi proses ini ialah pengaruh masa dan pengaruh luas permuakan biji yang terdedah kepada proses pengekstrakan. Suhu dan tekanan dikekalkan kepada 100 ^{o}C and 1 bar, dan masa optima untuk proses pengekstrakan akan dapat dikenalpasti daripada eksperimen ini. Komposisi utama minyak ini ialah Thymoquinone iaitu sebanyak 50 % daripada jumlah keseluruhan komposisi minyak. Oleh yang demikian, kewujudan komposisi ini boleh dikatogorikan sebagai karaktor utama kepada minyak yang akan dianalisa menggunakan Cecair Kromatogarm Keupayaan Tinggi (HPLC).

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CHAPTER 1

INTRODUCTION

1.0 Extraction

Extraction is phenomenon that can be defined as the process of separating desired components from a material. There are many types of extraction such as solid-liquid extraction and liquid-liquid. There are also a lot of extraction methods. Some of them are steam distillation, hydro distillation, solvent extraction, carbon dioxide extraction and cold pressing. The main objective in the extraction process is use to extract the essential oil. The main resources of essential oils are plants. In this research, the extraction will be conduct by using hydro distillation technique to extract essential oil from *Nigella Sativa seeds*.

1.1 Essential Oil

Essential oils or Volatile oils are the odorous principles found in various plant parts. It can be found in the bark of the plant, the flower of the plant or even in the seeds of the plant. Essential oils are oils that are found in bags inside these plants cell. These oils can be free from the bags and extracted using some of methods mentioned above. When they are exposed to air at ordinary temperatures they evaporate, therefore they are

called volatile oils, ethereal oils, or essential oils. The last term is applied because the oils represent the "essences" or odor constituents of the plants.

As a result, they are usually colorless, particularly when fresh, but with age they may oxidize and resinify, thus becoming darker, but sometimes the color of the oil is same with the material color. Therefore, storage should be in a cool, dry place, tightly stoppered, preferably full in amber glass containers. These essential oils have many uses. Most essential oils are produced for the perfume industry and minute amounts are used for flavors prepackaged foods. Besides that some essential oils that are produced from plants have high medical values in them like *Nigella Sativa* seeds.

1.2 Research Background / Problem Statement

In this research, Hydro Distillation Unit was used to get the *Nigella Sativa* essential oil. To obtain a series of high quality extraction from *Nigella Sativa*, the factors that influence the rate of extraction was study to get high quality of essential oil.

It is appropriate to improve the traditional hydro distillation method because of the energy wasting. The extraction using traditional hydro distillation method cannot give the highest purity and quality of Nigella Sativa Essential Oil. It is hard to determine the exact amount of the solvent. So the aiming of the research is to get the essential oil by using Rotary Evaporator representative the modern hydro distillation method. Figure 1.1 and 1.2 below show the traditional and modern hydro distillation technique[11] .

Figure 1.1: The Traditional Hydro Distillation Unit

Figure 1.2: The Modern Hydro Distillation Unit (rotary evaporator)

The Government wants to nurture the Malaysian herbal industry to be a leading international player. Therefore, the government had organized a lot of campaign and the latest one is "Celik Herba 2006 Campaign" and it was held on 19th to 20th September 2006 with FRIM collaboration. The local herbal industry is growing at an annual rate of between 15% and 20% and has a market value estimated at RM14 billion and will reach a size of RM24 billion by 2012[13].

Nowadays, in Malaysia, essential oil and oleoresin is gaining popularity as an herbal medication as it gave a lot of benefit to overcome the disease. It is because our people start to realize the important of healthy awareness. Hence, *Nigella Sativa* essential oil has a clear commercial value. So the identification of the constituents of *Nigella Sativa* has been carried out extensively over the last 10 years. Based on the knowledge on the constituent of our local *Nigella Sativa* and the potential commercial value essential oil as a whole, it is only appropriate if KUKTEM could further the research into producing and commercialize a new product from these invaluable herbs.

1.3 The Objective of the Research

To obtain essential oil from *Nigella Sativa* plant source using hydro distillation technique, competitive in terms of quantity and cost to essential oil produced by traditional methods, by investigating and understanding steam distillation extraction process.

1.4 The Scopes of the Research

To achieve the objective, scopes have been identified in this research. The scopes of this research are listed as below:-

1. To study the factor of influences of rate of extraction
    - Time
    - Surface area
2. To study the product analysis using HPLC

CHAPTER II

LITERATURE REVIEW

2.0 Overview of Nigella Sativa

Nowadays, the consumption of herbal based products are getting a wide spread acceptance among consumers because of the numerous beneficial therapeutic impacts they could give to our body and indirectly helps us sustaining a healthy condition. Although artificial and synthetic drugs are common in the market to combat a lot of chronic diseases, but these medications usually have negative effects to our body. Nowadays, people start to realize the differences between traditional and modern medication technique. Therefore, products from herbs, especially the Jintan Hitam (*Nigella Sativa)* are the right choice in treating certain kinds of ailments or diseases without introducing side effects to our body if consumed accordingly based on scientific findings and research. This is where our herbal products comes in which are confidently introduced to the public and backed by scientific research and findings from local and overseas scientist to prevent, reduce or to the extent in aiding the cure of certain types of chronic diseases or ailments.

The scientific term of Nigella Sativa have other synonyms such as Nigella damascena, Nigella ciliaris, Nigella arvensis and Nigella hispanica .Nigella Sativa is also known by its vernacular names such as Black Cumin (English), Cheveux de Vénus (France), Jinten hitam (Indonesia), Kalaunji (Punjabi), Habbet as-suda (Arabic), Jintan hitam (Malaysia), Hak jung chou (Chinese, Cantonese) and Thian dam (Thailand)[3]. This herb (Jintan Hitam known in Malaysia) is mentioned in the Quran, but today it is well known not only in Central and South Asia, but also in Western, its main application

area is Turkey, Lebanon and Iran. From Iran, nigella usage has spread to Northern India, particularly Punjab and Bengal

2.1 Characteristic of Nigella Sativa

Nigella Sativa is an annual flowering plant, native to southwest Asia. It grows to 20-30 cm tall, with finely divided, linear (but not thread-like) leaves. The flowers are delicate, and usually coloured pale blue and white, with 5-10 petals. The fruit is a large and inflated capsule composed of 3-7 united follicles, each containing numerous seeds. The seed is used as a spice [12]. Figure 2.0, 2.1 and 2.2 below show the flower, bud and the seeds of *Nigella Sativa*.

Figure 2.0: Nigella Sativa Flower

Nigella Sativa yield a seed capsule with five compartments each topped by a spike. The compartments open when dried to disperse the seeds. Nigella is native to western Asia where it grows both wild and cultivated. India, Egypt and the Middle East also cultivate it [12].

Figure 2.1: Nigella Sativa Unripe capsule

Nigella seeds are small, matte-black grains with a rough surface and an oily white interior. They are roughly triangulate, 1 1/2 - 3 mm (1/16 to 1/8 in) long. They are similar to onion seeds. The seeds have little bouquet, though when they are rubbed they give off an aroma reminiscent of oregano. It is also slightly bitter and peppery with a crunchy texture [12].

Figure 2.2: Nigella Seeds

2.2 Main constituents of Nigella Sativa

The seeds contain numerous esters of structurally unusual unsaturated fatty acids with terpene alcohols (7%); furthermore, traces of alkaloids are found which belong to two different types: isochinoline alkaloids are represented by nigellimin and nigellimin-*N*-oxide, and pyrazol alkaloids include nigellidin and nigellicin [14].

In the essential oil (avr. 0.5%, max. 1.5%), thymoquinone was identified as the main component (up to 50%) besides *p*-cymene (40%), α-pinene (up to 15%), dithymoquinone and thymohydroquinone. Other terpene derivatives were found only in trace amounts: Carvacrol, carvone, limonene, 4-terpineol, citronellol. Furthermore, the essential oil contains significant (10%) amounts of fatty acid ethyl esters. On storage,

thymoquinone yields dithymoquinonene and higher oligocondensation products (nigellone)[3].

The seeds also contain a fatty oil rich in unsaturated fatty acids, mainly linoleic acid (50 – 60%), oleic acid (20%), eicodadienoic acid (3%) and dihomolinoleic acid (10%) which is characteristic for the genus. Saturated fatty acids (palmitic, stearic acid) amount to about 30% or less. Commercial nigella oil ("Black Seed Oil", "Black Cumin Oil") may also contain parts of the essential oil, mostly thymoquinone, by which it acquires an aromatic flavour [3].

2.3 Uses and Benefit of Nigella Sativa

The seeds of the Nigella sativa plant are black in color and look something like sesame seeds. Both the seeds and oil from the seeds are used as a nutritional supplement. Black cumin seed is considered to have a number of beneficial properties when used as part of an overall holistic health program. Many studies show that, while black cumin seed is effective by itself, it is particularly potent when combined with other herbs in regimens used to treat specific ailments.

Black cumin seed (also referred to simply as "black seed") has been used as a nutritional supplement for centuries. Ancient traditions document the use of black cumin seed as an energy source, perhaps because of its rich nutritional value. The seeds are still believed to increase heat in the body, making metabolism more efficient. As a nutritional supplement in modern times, black cumin seed is used to treat respiratory conditions like bronchitis, asthma and emphysema. In addition, it is used to support stomach and

intestinal health as well as kidney and liver function. Black cumin seed is thought to have antihistamine-like properties that make it useful in treating congestion, and it is widely used as a general tonic to boost immune function and to help prevent cancer. Several skin conditions can be treated with black cumin seed, and it is also used to enhance circulation [5].

2.4 Overview of Separation Process

Separations are extremely important in Chemical manufacture. Separation processes are any set of operation that separate solutions of two or more components into two or more product that differ in composition. These may either removed a single components from a mixture or separate a solution into its almost pure components. This can be done by exploiting chemical and physical property differences between the substances through the used of a separating agent. There are three types of separation processes. 1st is gas –liquid separation, 2nd is liquid –liquid separation and 3rd is solid –liquid separation. In this research, solid-liquid separation processes will be conduct to separate the Nigella Sativa essential oil from the seeds [15].

2.5 Essential Oil Extraction Process

There are a lot of methods to extract the essential oils; the vast majority of true essential oils are produced by distillation. There are different processes used such as steam distillation, hydro distillation, cold pressing, vapor-cracking, turbo-extractor and many other solvent extractors. Hydro Distillation method will be use in this research because it's very efficient and easy to conduct.

2.5.1 Hydro distillation

Hydro distillation is used in the manufacture and extraction of essential oils. The botanical material is immersed in the water then being boiled with the water. The hot water helps to release the aromatic molecules from the plant material since the hot water forces to break the pockets in which the oils are kept in the plant material. The molecules of these volatile oils then escape from the plant material and evaporate into the steam.

The temperature of the process needs to be carefully controlled - just enough to force the plant material to let go of the essential oil, yet not too hot as to burn the plant material or the essential oil. The steam which then contains the essential oil is passed through a cooling system to condense the steam, which form a liquid from which the essential oil and water is then separated.

During distillation, only very tiny molecules can evaporate, so they are the only ones, which leave the plant. These extremely small molecules make up an essential oil. Oils containing more of the smallest, and therefore most volatile of these tiny molecules, are termed ‘top notes’ in the perfumery world; those containing more of the heaviest and least volatile of the tiny molecules are called ‘base notes’. Those in between are known as middle notes[15].